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(21) International Application Number: PCT/US((22) International Filing Date: 10 May 2000 ((30) Priority Data: 60/133,571 11 May 1999 (11.05.99) 09/568,187 9 May 2000 (09.05.00) (71) Applicant: PPG INDUSTRIES OHIO, INC. [US/U	10.05.0 t	BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA,
West 143rd Street, Cleveland, OH 44111 (US). (72) Inventors: MCCOLLUM, Gregory, J.; 5130 Bronw, Gibsonia, PA 15044 (US). HAUSER, Brian, Chelstead Way, Murrysville, PA 15668 (US). Joseph, T.; 420 Englewood Drive, Pittsburgh, P (US). GRAY, Ralph, C.; 137 West Cruikshar Butler, PA 16001 (US). (74) Agents: UHL, William, J.; PPG Industries, Inc., 6	T.; 40 VALK PA 152 nk Roa	14 Published O, With international search report. 37 Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.
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PROCESS FOR THE TREATMENT OF CONTINUOUS COILS OF SHEET METAL AND PRODUCTS PREPARED THEREBY

BACKGROUND OF THE INVENTION

The present invention relates to an improved process for the treatment of continuous coils of sheet metal produced in a steel mill.

The production of light gauge steels for end uses ranging from architectural construction to automobiles is well known. 15 The coils of steel are produced on a cold rolling mill line to the preferred thickness and trimmed or slit to the required width. The metal may be oiled and used directly as cold rolled steel or it may be further coated with a relatively thin layer of a second metal on a continuous galvanizing line. 20 The galvanizing process may be performed in a bath of molten metal, typically with a zinc alloy, or it may be performed electrolytically with essentially pure zinc or with a codeposit of zinc with a second metal such as iron. An annealing process may also be performed on the zinc coated 25 steel sheet to form an alloy with the steel substrate. The thus coated steel coil may then be oiled for storage or shipment to a customer for subsequent metal working operations. Eventually the oil must be removed by the customer using acid or alkaline cleaners before application of 30 passivating coatings ("pretreatment" coatings for adhesion and corrosion inhibition) or decorative coatings during manufacturing of the metal piece into a desired end product. Residues from the oil and cleaners may remain on the metal surface after cleaning and rinsing, inhibiting the active 35 ingredients in the passivating coatings from effectively bonding with metal atoms at the metal surface, thus diminishing the protective properties thereof.

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Hot dip galvanized sheet steel may also be passivated on the galvanizing line for resistance to white rust in storage, typically with a hexavalent chromium product. This is undesirable due to the toxicity of chromium, and the unsuitability as a base for subsequent coating.

It would be desirable to provide an improved process, without the use of chromium, for treating metals at the steel mill before application of protective lubricant, in order to enhance the corrosion protection of the metals as they are manufactured into end use products.

SUMMARY OF THE INVENTION

In accordance with the present invention, an improved process for treating a metal coated sheet steel is provided, the improvement comprising the following steps:

- a) contacting a surface of the metal coated sheet steel with a Group IIIB or IVB metal compound or mixture thereof dissolved or dispersed in a carrier medium; followed by
- b) contacting the surface of the metal coated sheet steel 20 from step (a) with a protective lubricant.

Another aspect of the present invention is a treated metal coated sheet steel formed in accordance with the process outlined immediately above.

Another aspect of the present invention is a coated metal sheet steel comprising: (a) a metal substrate; (b) a coating comprising a Group IIIB or IVB metal compound or mixture thereof formed upon at least a portion of a surface of the metal substrate; and (c) a protective lubricant coating formed upon at least a portion of the coating.

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DETAILED DESCRIPTION

Unless otherwise indicated, all numbers expressing quantities of ingredients or reaction conditions used in the specification and claims are to be understood as modified in 5 all instances by the term "about".

The process of the present invention is typically used to produce sheet metals such as steel coated with zinc metal, zinc compounds or zinc alloys such as electrogalvanized steel, hot-dipped galvanized steel, Galvannealed steel, and steel 10 plated with zinc alloy. Also, aluminum alloys, aluminum clad steel and aluminum alloy clad steel may be used. The shape of the metal substrate is an elongated strip wound about a spool in the form of a coil. The thickness of the strip preferably ranges from about 0.254 to about 3.18 millimeters (mm) (about 15 10 to about 125 mils), and more preferably about 0.3 mm, although the thickness can be greater or less, as desired. The width of the strip generally ranges from about 30.5 to about 183 centimeters (about 12 to about 72 inches), although the width can vary depending upon its intended use.

After coating the steel substrate, such as by electro galvanizing or hot dip galvanizing, and cooling the metal surface typically to a temperature of 100-300°F (37-149°C) and prior to the application of a protective lubricant such as oiling of the metal, the surface of the sheet metal is 25 contacted with a Group IIIB or IVB metal compound.

20

The Group IIIB or IVB metal compound is typically dispersed or dissolved in a carrier medium such as a aqueous medium, vapor, or aerosol. The solution or dispersion may be applied to the metal at a carrier medium temperature of about 30 ambient to 150°F (ambient to 65°C) by known application techniques, such as roll coating, dipping or immersion, spraying, intermittent spraying, dipping followed by spraying or spraying followed by dipping. The vapor or aerosol containing the Group IIIB or IVB metal may be applied by 35 passing the continuous strip of metal through a chamber in which the vapor is present. Treatment times are typically

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short, from a few seconds up to a minute, depending on the speed of the sheet metal line, and the type of coater being used.

The Group IIIB or IVB metals referred to herein are those elements included in such groups in the CAS Periodic Table of the Elements as is shown, for example, in the Handbook of Chemistry and Physics, 63rd Edition (1983).

Preferred Group IIIB and IVB metal compounds are compounds of zirconium, titanium, hafnium, yttrium and cerium 10 and mixtures thereof. Typical zirconium compounds may be selected from hexafluorozirconic acid, alkali metal and ammonium salts thereof, ammonium zirconium carbonate, zirconyl nitrate, zirconium carboxylates and zirconium hydroxy carboxylates such as hydrofluorozirconic acid, zirconium 15 acetate, zirconium oxalate, ammonium zirconium glycolate, ammonium zirconium lactate, ammonium zirconium citrate, and mixtures thereof. Hexafluorozirconic acid is preferred. An example of an yttrium compound is yttrium nitrate. An example of the titanium compound is fluorotitanic acid and its salts. 20 An example of the hafnium compound is hafnium nitrate. An example of the cerium compound is cerous nitrate. The Group IIIB or IVB metal compound is present in the carrier medium in an amount of 10 to 5000 ppm metal, preferably 100 to 1000 ppm metal.

For aqueous carriers, the pH of the solution with dispersion usually ranges from 2.0 to about 7.0 and is preferably about 3.5 to about 5.5. The pH may be adjusted using mineral acids such as hydrofluoric acid, fluoroboric acid, phosphoric acid, and the like, including mixtures thereof; organic acids such as lactic acid, acetic acid, citric acid, or mixtures thereof; and water soluble or water dispersible bases such as sodium hydroxide, ammonium hydroxide, ammonia, or amines such as triethylamine, methylethyl amine, or mixtures thereof.

35 In one embodiment of the invention, the solution or dispersion further contains a film forming resin. Suitable

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resins include reaction products of one or more alkanolamines and an epoxy-functional material containing at least two epoxy groups, such as those disclosed in U. S. Patent 5,653,823. Preferably, such resins contain beta hydroxy ester, imide, or 5 sulfide functionality, incorporated by using dimethylolpropionic acid, phthalimide, or mercaptoglycerine as an additional reactant in the preparation of the resin. Other suitable resins include water soluble and water dispersible polyacrylic acids as disclosed in U. S. Patents 3,912,548 and 10 5,328,525; phenol-aldehyde resins as described in U. S. Patent 5,662,746; water soluble polyamides such as those disclosed in WO 95/33869; copolymers of maleic or acrylic acid with allyl ether as described in Canadian Patent application 2,087,352; and water soluble and dispersible resins including epoxy 15 resins, aminoplasts, phenol-formaldehyde resins, tannins, and polyvinyl phenols as discussed in U. S. Patent 5,449,415. Another suitable resin is the reaction product of an epoxycontaining polymer or oligomer (polyepoxide) with a dialkanolamine and a hydroxy acid. Examples of suitable 20 epoxy-containing polymers or oligomers include polyglycidyl ethers of polyhydric phenols such as the polyglycidyl ether of Bisphenol A. The preferred polyglycidyl ether is the diglycidyl ether of Bisphenol A.

Examples of dialkanolamines include those which contain
25 up to four carbon atoms such as diisopropanolamine, diethanolamine, di(2-hydroxybutyl)amine, and N-(3-hydroxypropyl)
ethanolamine. Diethanolamine is preferred.

Examples of hydroxy acids include dimethylolpropionic acid, which is preferred, trimethylolpropionic acid,

pentaerythritol, malic acid, lactic acid, glycolic acid, gluconic acid, glucuronic acid, citric acid, 3-hydroxypivalic acid, and salicylic acid.

The reaction product may be prepared as follows: the epoxy-containing polymer or oligomer is added to a suitable reaction vessel with an organic solvent. Suitable solvents include glycol ethers such as ethylene glycol methyl ether and

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propylene glycol methyl ether. The mixture is heated to a temperature of about 50°C, the amine and hydroxy acid are added, and the mixture allowed to exotherm to a temperature of about 90 to 100°C.

The mole ratio of the epoxy-containing polymer or oligomer to hydroxy acid to amine is from 0.6 to 5.0:0.05 to 5.5:1, preferably 1.5 to 2.5:1.0 to 2.0:1.

The reaction product may then be thinned with additional organic solvents and/or water preferably containing an acid which can form the carrier medium for the reaction product. Examples of other organic solvents include alcohols with up to about 8 carbon atoms such as methanol, isopropanol, and the like, additional glycol ethers such as the monoalkyl ethers of ethylene glycol, diethylene glycol, or propylene glycol, and the like. Water, containing sulfamic acid, is the preferred ingredient in the carrier medium. When present, the water dispersible organic solvents are typically used in amounts up to about twenty (20) percent, preferably ten (10) percent by volume, based on the total volume of the carrier medium with water being the remaining.

When present, the film forming resinous binder is in the carrier medium of the present invention in an amount of 0.005% to 30% based on the total weight of the ingredients in the medium, and the weight ratio of the resin to Group IIIB or IVB metal or metal compound is from 2.0 to 10.0:1, preferably 3.0 to 5.0:1 based on metal.

Other optional materials in the carrier medium include surfactants that function as defoamers or substrate wetting agents. Anionic, cationic, amphoteric, or nonionic surfactants may be used. Compatible mixtures of such materials are also suitable. Surfactants are typically present at levels up to about 1 percent, preferably up to about 0.1 percent by volume, and wetting agents are typically present at levels up to about 2 percent, preferably up to about 0.5 percent by volume, based on the total volume of carrier medium.

The film coverage of the residue of the pretreatment coating composition generally ranges from about 1 to about 1000 milligrams per square meter (mg/m²), and is preferably about 10 to about 400 mg/m². The thickness of the pretreatment coating can vary, but is generally less than about 1 micrometer, preferably ranges from about 1 to about 500 nanometers, and more preferably is about 10 to about 300 nanometers.

After contact with the Group IIIB or IVB metal compound,

the sheet metal is coated with a protective wet or dry
lubricant for shipping or storage. Such lubricants may be any
of those used conventionally in the art. An example of a wet
lubricant is oil. U. S. Patent No. 5,229,450 describes a
suitable dry lubricant.

Not intending to be bound by any theory, the inventors believe that by forming the metal according to the process of the present invention, a stronger chemical interaction between metal atoms at the metal surface and the Group IIIB or IVB metal compound is possible because the virgin metal surface is free of residues of oil and alkaline or acidic cleaners that may inhibit chemical reaction, thus providing enhanced corrosion protection of the finished metal product.

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WE CLAIM:

- 1. In a process for treating a metal coated steel sheet, the improvement comprising the following steps:
- (a) contacting a surface of the metal coated sheet steel with a Group IIIB or IVB metal compound or mixture thereof dissolved or dispersed in a carrier medium; followed by
 - (b) contacting the surface of the metal coated sheet steel from step (a) with a protective lubricant.

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- 2. The process of claim 1 wherein the Group IIIB or IVB metal compound is present in an aqueous carrier medium, vapor or aerosol.
- 3. The process of claim 2 wherein the metal surface is contacted with the Group IIIB or IVB metal compound in the aqueous carrier medium at a carrier medium temperature of about ambient to 150°F (ambient to 65°C).
- 20 4. The process of claim 2 wherein the metal surface is contacted with the Group IIIB or IVB metal compound by roll coating.
- 5. The process of claim 2 wherein the metal surface is contacted with the Group IIIB or IVB metal compound as an aerosol or a vapor.
 - 6. The process of claim 1 wherein the Group IIIB or IVB metal compound is a zirconium compound.

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- 7. The process of claim 6 wherein the zirconium compound is present in the aqueous medium in an amount of 10 to 5000 ppm Zr.
- 35 8. The process of claim 2 wherein the aqueous medium further comprises a resinous binder.

-----MATTER DESCRIPTION----- Taiwanese Trademark Appln. No. 94026676 Mark: EXTRON

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*CLIENT INFORMATION RGB SYSTEMS, INC.	JOINT BILL: 11845	*CLIENT ADDRESS*	1230 SOUTH LEWIS STREET ANAHEIM, CA 92805	*MATTER INFORMATION PHONE: 780-4088	REFERRED BY:	STATUS: OP	DATE OPENED: 06/01/2005	DATE CLOSED:	LAST RATE: 07/21/2004	HOLD FEES:	HOLD COSTS:	TRUST RET ACCT:	17	TRADEMARK	01/01/00	-3·	

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- 9. The process of claim 8 wherein the resinous binder comprises a reaction product of an epoxy group-containing polymer or oligomer, a hydroxy functional acid and a 5 dialkanolamine.
 - 10. The process of claim 9 wherein the epoxy group-containing polymer or oligomer is a polyglycidyl ether of a polyhydric phenol.

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- 11. The process of claim 9 wherein the epoxy group-containing polymer or oligomer is the diglycidyl ether of Bisphenol A.
- 15 12. The process of claim 9 in which the dialkanolamine is diethanolamine.
 - 13. The process of claim 9 in which the hydroxy functional acid is dimethylolpropionic acid.

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14. The process of claim 9 wherein the epoxy group-containing polymer or oligomer, hydroxy functional acid, and dialkanolamine are reacted in a 0.6 to 5.0:0.05 to 5.5:1 mole ratio.

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- 15. The process of claim 9 wherein the medium is substantially free of chrome.
- 16. The process of claim 1 wherein the metal is zinc 30 coated steel.
 - 17. A treated metal coated sheet steel formed in accordance with the process of claim 1.

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- 18. A coated metal sheet steel, comprising:
- (a) a metal substrate;
- (b) a coating comprising a Group IIIB or IVB metal compound or mixture thereof formed upon at least a portion of
 5 a surface of the metal substrate;
 - (c) and a protective lubricant coating formed upon at least a portion of the coating.
- 19. The coated metal sheet steel of claim 18 wherein the 10 Group IIIB or IVB metal compound is a zirconium compound.
 - 20. The coated metal sheet steel of claim 18 wherein the coating comprising a Group IIIB or IVB metal compound or mixture thereof is substantially free of chrome.

21. The coated metal sheet steel of claim 18 wherein the metal is zinc coated steel.

DATE: 04/11/06 13:45:23 PRO FORMA STATEMENT AS OF 033106 FOR FILE (T34843NZ1) T34843NZ1

New Zealand Trademark Application *-----MATTER DESCRIPTION----* Mark: EXTRON

--SUPERVISING--J HOLMAN --BILLING--J HOLMAN --ORIGINATING--J HOLMAN --CLIENT NUMBER--11845 *----CLIENT INFORMATION----* RGB SYSTEMS, INC.

FOLLOWUP TEMPLATE: ER BILL TEMPLATE: EJ -----CLIENT ADDRESS----* JOINT BILL: 11845

-----MATTER ADDRESS----

-----MATTER INFORMATION----* 1230 SOUTH LEWIS STREET ANAHEIM, CA 92805 RGB SYSTEMS, INC.

PHONE: 780-4088 REFERRED BY:

CONTACT: PHONE:

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· INTERNATIONAL SEARCH REPORT

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	ocumentation searched (classification system followed by classification	on symbols)	
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"A" docum	categories of cited documents: ment defining the general state of the art which is not addresd to be of particular relevance r document but published on or after the international	"T" later document published after the inter- or priority date and not in conflict with cited to understand the principle or the invention "X" document of particular relevance; the or	the application but early underlying the stained invention
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	12 September 2000	16/10/2000	
Name and	d mailing address of the ISA European Patent Office, P.B. 5818 Patentiaan 2 NL - 2280 HV Rijswijk	Authorized officer	
	Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	De Anna, P	

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CLIENT INFORMATION RGB SYSTEMS, INC. JOINT BILL: 11845	CLIEN 11845 BILL TEMPLATE: EJ	-CLIENT NUMBERORIGINI 11845 J. HOLMAN EJ. FOLLOWUP TEMPLATE: ER	ORIGINATING JCH J. HOLMAN LATE: ER	BILLING JCH J HOLMAN	SUPERVISING JCH J HOLMAN
CLIENT ADDRESS RGB SYSTEMS, INC. 1230 SOUTH LEWIS STREET ANAHEIM, CA 92805 *MATTER INFORMATION* PHONE: 780-4088 REFERRED BY:		**MATTER ADDRESS-PHONE:	*		· · · · · · · · · · · · · · · · · · ·
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INTERNATIONAL SEARCH REPORT

information on patent family members

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